BARIUM POWDER GETTER PRODUCTION METHOD

Original Filed April 22, 1965 2 Sheets-Sheet 1

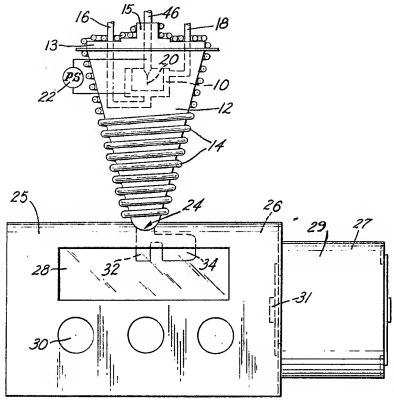

Fig. 1.

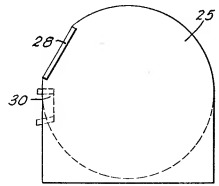

Fig. 2.

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BARIUM POWDER GETTER PRODUCTION METHOD

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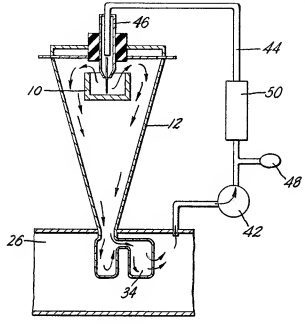

Fig. 3.

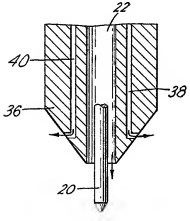

Fig. 4.

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BARIUM POWDER GETTER PRODUCTION METHOD

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3 Claims. (Cl. 75—.5)

ABSTRACT OF THE DISCLOSURE

Barium is vaporized with an electric arc in an inert gas such as helium, and the so vaporized barium particles are blown away from the area of the arc and cooled in order to prevent their coagulating into a mass having a size over 200 Angstroms.

This is a continuation of Ser. No. 449,980, filed Apr. 22, 1965, and now abandoned.

This invention relates to the manufacture of getters, and more particularly to the production of barium powder that is suitable for use as a gettering material.

Powdered barium is being increasingly used as a gettering material for the maintenance of vacuums. In double walled vacuum insulated cryogenic containers, for example, powdered barium is used to maintain the vacuum between the container walls by combining with or adsorbing residual or leaking gases other than the noble gases in the vacuum space. Such powder will normally be held within the vacuum space in a porous bag. Or as another example, baruim powder can be used to maintain the vacuum in the vacuum tubes of electronic equipment.

In such uses, the particle size of the barium powder should be no greater than 200 Angstroms in diameter in order to be effective as a gettering material. This leads to the problem of mass producing the small particle size powder.

It is, therefore, the principal object of the present invention to provide a method and apparatus for economically producing large amounts of barium powder that is suitable as a gettering material.

Briefly, according to the invention, an electric arc is used to heat and vaporize solid barium in an atmosphere that is inert with respect to barium. The so-vaporized barium is then cooled and collected in a suitable receptacle in such atmosphere. The preferred atmosphere is helium gas.

In the drawings:

FIGURE 1 is a fragmentary view mainly in front elevation of apparatus illustrative of the invention;

FIGURE 2 is an end view of such apparatus;

FIGURE 3 is a flow diagram of the invention; and

FIGURE 4 is a fragmentary vertical section of the preferred torch.

Referring to FIGURES 1 and 2, crucible **10** which holds the barium to be vaporized is contained within an inverted conical housing **12** having a cover **13**. Housing **12** is cooled by passing water through coil **14**. Crucible **10**, which is constructed of copper, is also cooled by passing cooling water from conduit **16** into cooling passages (not shown) in the crucible and thence out conduit **18**.

An arc is established and maintained between electrode **20** of the torch **46** mounted in cover **13**, and the crucible by connecting a suitable electrical power supply **22** to the electrode and the crucible. Thus, the arc operates in the transferred mode. In operation, direct current with straight polarity (electrode negative) is preferred. However, it should be understood that direct current with reverse polarity, as well as alternating current could also be used.

Housing **12** has a bottom opening or exit **24** at its inverted apex. At such exit, the housing is leak tightly connected to cylindrical casing **25** providing a chamber **26**. The front wall of the casing **25** is provided with an upper window **28**, and a plurality of round openings **30**. Although not shown, rubber gloves are leak tightly connected to each of the openings **30**, so that an operator can work inside the chamber **26** while observing the work through the window.

An auxiliary casing **27** is attached to the end of casing **25** to provide an auxiliary chamber **29**. From the chamber **26**, an entrance into an auxiliary chamber **29** is achieved through a door **31**. Extending from the bottom opening in the apex of the conical housing is a collector jar **32** for larger particles of barium powder. Extending from the jar **32** is a porous receptacle **34**, such as a bag of cloth or suitable material for collecting the finer powder, useful as gettering material.

In operation, a charge of solid barium metal is placed in the crucible **10**, the charge being heated and melted by the arc **20**. As the charge becomes vaporizer, the flow of helium gas emanating from the vicinity of the electrode for the arc, forces the so-vaporized barium up and away from the heated zone close to the walls of the conical housing **12**, so as to cool the vaporized particles. The helium gas flow then carries the cooled particles down through the exit **24** of the housing **12** and thence into the porous receptacle **34**. The gas is then recirculated back to the electrode torch **46**.

Any large particles not carried by the flowing gas into the porous receptacle will drop into the jar **32**. Once the receptacle **34** is filled, the operator disconnects it through the openings **30** and empties the powdered barium into separate tubes, seals the tubes, and then places them in the auxiliary chamber **29**. This procedure of using the auxiliary chamber preserves the needed helium atmosphere in the main chamber **26** by preventing the inspiration of other gases into the main chamber.

The tubes with the barium powder are then taken from the auxiliary chamber, attached to a manifold, and evacuated to a pressure of about 20 microns of mercury. The auxiliary chamber is then purged with helium gas, and the operation repeated as necessary.

FIGURES 3 and 4 illustrate the critical gas flow pattern necessary for achieving the desired small powder size of the barium. Once the barium charge, held within the crucible **10**, starts to vaporize, it is necessary that the vapor particles be forced away from the area of the arc and be cooled before they have a chance to coagulate into a mass unsuitable as gettering material. Thus, the preferred gas flow for carrying the barium vapor is as shown by the arrows.

To achieve this flow, a torch T constructed as shown in FIGURE 4 is preferred. As shown, there are two separate gas streams: (1) the flow of electrode gas down around the electrode **20** in the annulus **22** formed by the electrode and the nozzle or electrode holder **36**; (2) a second stream that is ejected substantially normal to the flow of electrode gas through ports **38** by way of annulus **40**. These combined gas flows force the vaporized barium up and out of the crucible, away from the electrode to be cooled. Thus, the vapor is rapidly cooled, and in addition, contamination of the electrode is prevented. As shown in FIG. 3, the gas flow proceeds down along the walls of the housing **12** through porous bag **34**, out of the chamber **26** to be recirculated by compressor **42** in conduit **44** that is connected to torch **46**. A pressure gauge **48** and a flowmeter **50** are connected to such conduit **44**.

The preferred gas for carrying out the invention is helium. However, other gases that are inert to barium can also be used.

The arc voltage for a given current has been found to be critical. Such voltage is dependent upon the standoff distance of the electrode from the melt; the greater the

distance, the greater the voltage. If the voltage is too low, the particle size becomes too large. On the other hand, if the voltage is too high, the arc concentration at the melt becomes smaller and production of the vapor particles drops off, but the particle size becomes finer. Thus, the arc voltage should be maintained within a range which is a balance of particle size versus production rate. For a current of 150 amperes, an arc voltage of 19 to 20 volts has been found to be optimum.

As a typical example of the operation of the process of the invention, two pounds of solid barium metal were placed in the crucible and heated and vaporized with an arc operating at 150 amperes at 20 volts. Helium gas at 200 c.f.h. was passed down around the electrode and 100–200 c.f.h. of such gas was passed out through the radial ports **38**. Under such conditions barium powder, suitable as gettering material, was produced at the rate of about 60 gm./hr. The powder had a gettering capacity of 29,000 micron-liters of N_2 gas capacity; the theoretical capacity of barium being 30,000 micron-liters of N_2 gas.

In the present process the metal barium is vaporized and subsequently condensed while gas-borne. The particular manner in which the vaporized barium is connected (gas flow pattern) to insure the small particle size is also new.

What is claimed is:

1. A method of making active barium powder gettering material comprising:

(A) Directing an electric arc at barium in an inert environment at above atmospheric pressure to heat and vaporize said barium;

(B) Flowing the vaporized barium upwardly in a first inert gas stream;

(C) Deflecting the upwardly flowing first inert gas-vaporized barium stream outwardly and away from the electric arc in a second inert gas stream so as to cool and condense the barium as powder; and

(D) Separating the barium powder and the first and second inert gases.

2. Method of making gettering material as defined by claim **1** in which the barium is vaporized by an electric arc in a crucible with a torch through which helium gas flows, first downwardly over the molten barium and then upwardly and outwardly over the rim of such crucible, and then downwardly in an inverted conical chamber the housing of which is externally cooled to condense the helium gas-borne barium into small particles that are discharged through an exit in the bottom of such chamber.

3. Method of making gettering material as defined by claim **2**, in which the helium gas is recirculated back to such torch after the barium particles are removed therefrom.

References Cited

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